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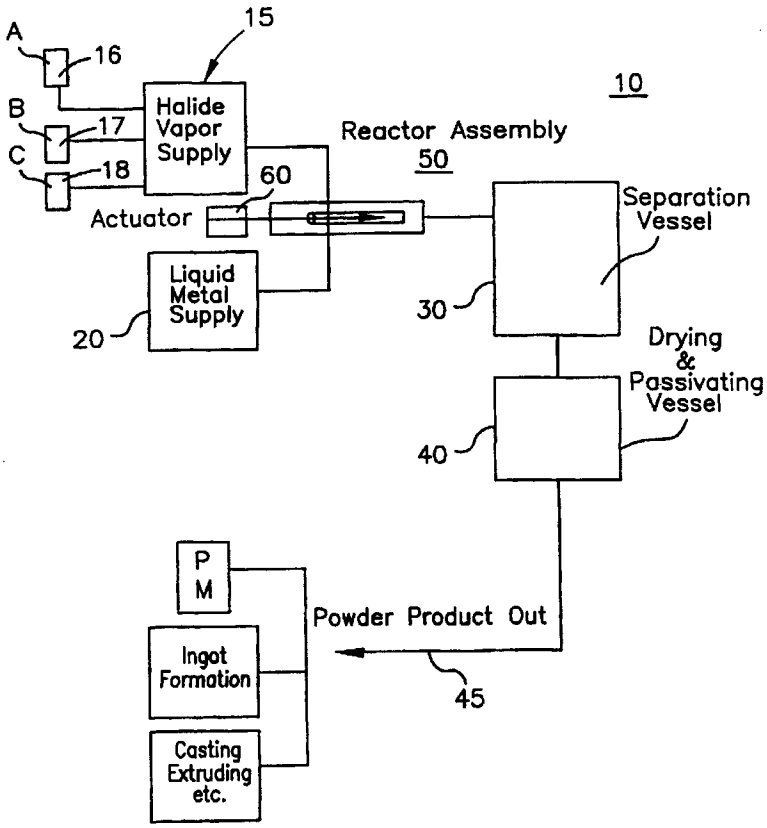
(54) Title: SAFETY MECHANISM



(57) Abstract: A safety mechanism used in the subsurface exothermic reduction of a halide vapor with a liquid reducing metal. A reactor for introducing halide vapor into the liquid metal causing an exothermic reaction forming a slurry of excess liquid metal and salt and a product powder. There is a source of halide vapor in fluid communication with said reactor and a source of liquid metal in fluid communication with the reactor. A porous plug is intermediate the source of the halide vapor and the source of liquid metal preventing liquid metal from infiltrating the source of halide vapor by forming a seal in said porous plug upon contact with liquid metal.

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Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

— *with international search report*

— *with amended claims and statement*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

SAFETY MECHANISM

RELATED APPLICATIONS

This application, pursuant to 37 C.F.R. 1.78(c), claims priority based on provisional application U.S. Provisional Application Serial No. 60/408,927 filed September 7, 2002.

BACKGROUND OF THE INVENTION

This invention relates to the Armstrong process as described in U.S. patents 5,779,761, 5,958,106 and 6,409,797, the disclosures of each of which is incorporated herein by reference. In the method described in the above-referenced patents, the halide vapor is introduced into the reductant liquid metal at a flow velocity which is equal to or greater than sonic in order to preclude the backup of liquid metal into the halide vapor supply. Such a backup could be catastrophic. This invention relates to an additional safety measure in the use of a porous plug such as porous metal mesh or sintered metal powder in the inlet line between the nozzle where the halide vapor flows into the liquid metal and the source of the halide vapor. A porous plug as previously described which has a material therein which swells upon contact with the reductant metal (sodium) would, upon contact with the liquid metal reductant (sodium), react and swell forming a seal in the line preventing any additional reductant (sodium) from being sucked into the halide supply.

Accordingly, this invention relates to a porous plug in the inlet line between the titanium tetrachloride boiler and the injection point 23 as illustrated in Fig. 2 of the '106 patent. The porous plug seals either with sodium chloride which solidifies in the plug, or the plug may have a material such as graphite which reacts with liquid reductant (sodium) to swell in the plug and form a seal or a combination of both. This invention provides an important safety feature since check valves are notoriously unreliable and because of the possibility that a catastrophic explosion could occur should the halide vapor boiler fail and a vacuum be created, thereby sucking sodium into the halide boiler and/or the halide supply.

SUMMARY OF THE INVENTION

Accordingly, an object of the present invention is to provide a safety device in a system for practicing the Armstrong Process.

Still another object of the invention is to provide a simplified, easy insert into the line between the halide vapor boiler and the reactor which precludes the back-up of liquid metal into the halide boiler.

Yet a further object of the invention is to provide a system for producing a powder by the subsurface exothermic reduction of a halide vapor with a liquid alkali metal or alkaline earth metal or mixtures thereof, comprising a reactor for introducing halide vapor into the liquid metal causing an exothermic reaction forming a slurry of excess liquid metal and salt and a product powder, a source of halide vapor in fluid communication with said reactor, a source of liquid metal in fluid communication with the reactor, and a porous plus intermediate the source of the halide vapor and the source of liquid metal preventing liquid metal from infiltrating the source of halide vapor by forming a seal in said porous plug upon contact with liquid metal.

Yet another object of the invention is to provide a powder produced by the system previously described having the additional safety feature herein described.

A final object of the present invention is to provide a solid product made from a powder produced by the use of the system disclosed herein.

The invention consists of certain novel features and a combination of parts hereinafter fully described, illustrated in the accompanying drawings, and particularly pointed out in the appended claims, it being understood that various changes in the details may be made without departing from the spirit, or sacrificing any of the advantages of the present invention.

BRIEF DESCRIPTION OF THE DRAWINGS

For the purpose of facilitating an understanding of the invention, there is illustrated in the accompanying drawings a preferred embodiment thereof, from an inspection of which, when considered in connection with the following description, the invention, its construction and operation, and many of its advantages should be readily understood and appreciated.

FIGURE 1 is a schematic representation of a system for practicing the present invention; and.

FIGURE 2 is a schematic representation of a needle valve assembly useful in the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring to Figure 1, there is shown a system 10 for the practice of the present invention including a supply of halide vapor 15 which in turn is in fluid communication with a plurality of liquid halide or solid halide materials, shown for purposes of illustration only as supplies 16, 17 and 18 for halide liquids or solids A, B and C respectively. The system 10 further includes a supply of liquid metal 20 which may be any alkali or alkaline earth metal or various mixtures thereof, sodium and magnesium being preferred with sodium being mostly preferred.

Similarly, with respect to the halide vapor supply 15, chlorides are preferred and more specifically, one or more of the chlorides of Ti, Al, Sb, Be, B, Ta, Zr, V, Nb, Mo, Ga, U, Re and Si.

There is further provided a separation vessel 30 in fluid communication with a reactor assembly 50, as will be described, and in fluid communication with a drying and passivating vessel 40. A powder product outlet 45 is in communication with the drying and passivating vessel 40, as will be described, the powder being either the final product or an intermediate product of the system and process of the invention.

The present invention and system 10 includes the reactor assembly 50, as seen in Fig. 2, which has an outer cylinder 51 having an exit portion 52 which may be of reduced diameter or of the same diameter as the remainder of the outer cylinder or conduit 51, as preferred.

The reactor assembly 50 serves to receive the halide vapor of the metal or ceramic to be produced and the liquid reducing metal and to introduce the halide vapor in a controlled fashion but at not less than sonic velocity subsurface of the reducing metal or into a stream of the reducing metal so that the temperature of the reaction is controlled, in part, by the excess of the reducing metal, all is taught in the above-referenced patents.

The reactor assembly 50 has one-half of a sealing ring 54 on the exit nozzle portion 52 to sealing engage another sealing ring (not shown) located in the vessel into which the exit portion 52 is positioned. The outer cylinder 51 also has a inlet nozzle portion 56 which terminates in an end 57. An actuator 60, either pneumatic or otherwise, as is known in the art, is in communication with the reactor assembly 50 and particularly the outer cylinder 51 as will be explained. The outer cylinder 51 also has

a pressure tap 62 which may be for the introduction of an inert gas such as argon or to vent the assembly 50, if required, or to monitor the pressure within the outer cylinder 51. Also provided is a reducing metal inlet 64, in the illustration a sodium inlet. Both the pressure tap 62 and the reducing metal inlet 64 extend through the outer cylinder 51 and are sealed thereto.

A sealing ring is made up of mating halves 66 and 67 intermediate the actuator 60 and the exit nozzle portion 52 of the reactor assembly 50. A halide inlet tap 69 extends into the inlet nozzle portion 56 of the outer cylinder 51 and is sealed downstream of the inlet 69 by means of the sealing rings 66, 67 and is in fluid communication with a housing 79 which may be generally cylindrical in shape and extends from the sealing half ring 66 through the outer cylinder 51 and terminates at an end 81 having a valve seat therein.

A needle valve 75 includes an elongated cylindrical shaft portion 76 having a conical shape valve portion 77 and another end 78 in communication with the actuator 60. The halide inlet 69 introduces halide vapor into the chamber formed by the inlet nozzle portion 56 of the outer cylinder 51 and enters the housing 79 by virtue of the communication between the end of the housing 79 and the sealing rings 66, 67. The sodium entering through sodium inlet 64 is on the outside of the housing 79 and completely fills the outer cylinder 51 and flows axially of the outer cylinder. The longitudinal axial movement of the needle valve 75 by means of the actuator 60 causes the conical end portion 77 to seat within a valve seat in the end 81 of the housing 79, it being apparent to those of ordinary skill in the art that the diameter of the valve seat in the end 81 must be smaller than the diameter of the shaft portion 76 of the needle valve 75. Valve seats 81 between 1/8 and 3/8 inch have been used with the appropriate change in shaft portion 76.

It is important that no sodium be able to back up through the valve seat in the end 81 into the halide vapor supply. That necessity is accomplished by using not less than sonic flow of the halide through reactor assembly 50 as taught in the referenced patents and is insured by a porous plug 70 in the line between the vapor supply or boiler 15 and the needle valve 50. As regards the actuator 60, it may be operated to move the shaft portion 76 axially of outer cylinder 51 to the right in Fig. 2, so that the conical portion 77 of the needle valve 75 begins to seat within the valve seat in the end

81, the amount or volume of halide vapor, such as titanium tetrachloride, introduced into the sodium or reducing metal inside the outer cylinder 51 is reduced or controlled permitting the operators of the system to vary the time and rate of delivery of the halide vapor. Another advantage of the needle valve 75 is that when the needle valve 75 is fully seated within the valve seat in the end 81, a vacuum may be drawn upstream of the nozzle or reactor assembly 50 before startup of the production of the metal by the exothermic reaction of the halide with the reducing metal.

Turning now to the porous plug 70, it may be any suitable porous or perforated metal, such as but not limited to stainless steel. The plug 70 may be apertured or it may be a mesh, the purpose being if a catastrophic failure in protection occurred, as liquid metal moved toward the halide supply or boiler 15, a reaction would occur in the plug 70 forming salt particles which would seal the vapor supply or boiler 15 from the liquid metal supply 20. In addition, the plug 70, not being heated, should form a heat sink cooling the reactants sufficiently to solidify the formed salt. Cooling fins (not shown) or other cooling mechanism, well known in the art, may be useful in conjunction with plug 70 to ensure no liquid metal reaches the vapor boiler or supply 15. The size of the plug 70 is not shown to scale and is within the skill of the art to determine the diameter and length required to prevent back-up of liquid metal into the vapor supply 15. Another aspect of the invention is to contact the interior of the plug 70 with a material which reacts with the liquid metal, but not the halide vapor, that swells and seals the plug. For sodium metal, graphite is acceptable as it swells upon contact with liquid sodium and would form an effective seal. Other materials are within the skill of the art to identify and employ.

It is seen that the powder product 45 discharged from the drying and passivating vessel 40 may be used as a product in and of itself or may be used in powder metallurgy to produce product or ingot or other means by which solid product is formed which also includes casting, extruding or other methods. Any solid product or object made from the powder 45 produced by the inventive system 10 is within the purview of the present invention.

While there has been disclosed what is considered to be the preferred embodiment of the present invention, it is understood that various changes in the details may be made without departing from the spirit, or sacrificing any of the advantages of the present invention.

WHAT IS CLAIMED IS:

1. A system for producing a powder by the subsurface exothermic reduction of a halide vapor with a liquid alkali metal or alkaline earth metal or mixtures thereof, comprising a reactor for introducing halide vapor into the liquid metal causing an exothermic reaction forming a slurry of excess liquid metal and salt and a product powder, a source of halide vapor in fluid communication with said reactor, a source of liquid metal in fluid communication with the reactor, and a porous plus intermediate the source of the halide vapor and the source of liquid metal preventing liquid metal from infiltrating the source of halide vapor by forming a seal in said porous plug upon contact with liquid metal.

2. The system of claim 1, wherein said plug is porous metal.

3. The system of claim 1, wherein said plug is metal mesh.

4. The system of claim 1, wherein said plug is sintered metal powder.

5. The system of claim 1, wherein said plug contains a substance reactive with said liquid metal sealing said plug upon contact by said liquid metal.

6. The system of claim 5, wherein said plug contains graphite.

7. The system of claim 1, wherein said source of liquid metal is Na or Mg.

8. The system of claim 1, wherein said source of liquid metal is Na.

9. The system of claim 1, wherein said source of halide is a chloride.

10. The system of claim 1, wherein said source of halide is a mixture of chlorides.

11. The system of claim 1, wherein said source of halide includes one or more of the chlorides of Ti, Al, Sb, Be, B, Ta, Zr, V, Nb, Mo, Ga, U, Re and Si.

12. The system of claim 11, wherein said liquid metal is Na.

13. A powder produced by the operation of the system of claim 1.

14. A solid product made from the powder of claim 13.

AMENDED CLAIMS

Received by the International Bureau 13 February 2004 (13.02.04) ;
Original claims 1 replaced by amended claims 1.

WHAT IS CLAIMED IS:

1. A system for producing a powder by the subsurface exothermic reduction of a halide vapor with a liquid alkali metal or alkaline earth metal or mixtures thereof, comprising a reactor for introducing halide vapor into the liquid metal causing an exothermic reaction forming a slurry of excess liquid metal and salt and a product powder, a source of halide vapor in fluid communication with said reactor, a source of liquid metal in fluid communication with the reactor, and a porous plug intermediate the source of the halide vapor and the source of liquid metal preventing liquid metal from infiltrating the source of halide vapor by forming a seal in said porous plug upon contact with liquid metal.

2. The system of claim 1, wherein said plug is porous metal.

3. The system of claim 1, wherein said plug is metal mesh.

4. The system of claim 1, wherein said plug is sintered metal powder.

5. The system of claim 1, wherein said plug contains a substance reactive with said liquid metal sealing said plug upon contact by said liquid metal.

6. The system of claim 5, wherein said plug contains graphite.

7. The system of claim 1, wherein said source of liquid metal is Na or Mg.

8. The system of claim 1, wherein said source of liquid metal is Na.

9. The system of claim 1, wherein said source of halide is a chloride.

10. The system of claim 1, wherein said source of halide is a mixture of chlorides.

11. The system of claim 1, wherein said source of halide includes one or more of the chlorides of Ti, Al, Sb, Be, B, Ta, Zr, V, Nb, Mo, Ga, U, Re and Si.

12. The system of claim 11, wherein said liquid metal is Na.

13. A powder produced by the operation of the system of claim 1.

STATEMENT OF ARTICLE 19

Claim 1 is amended to correct a typographical error.

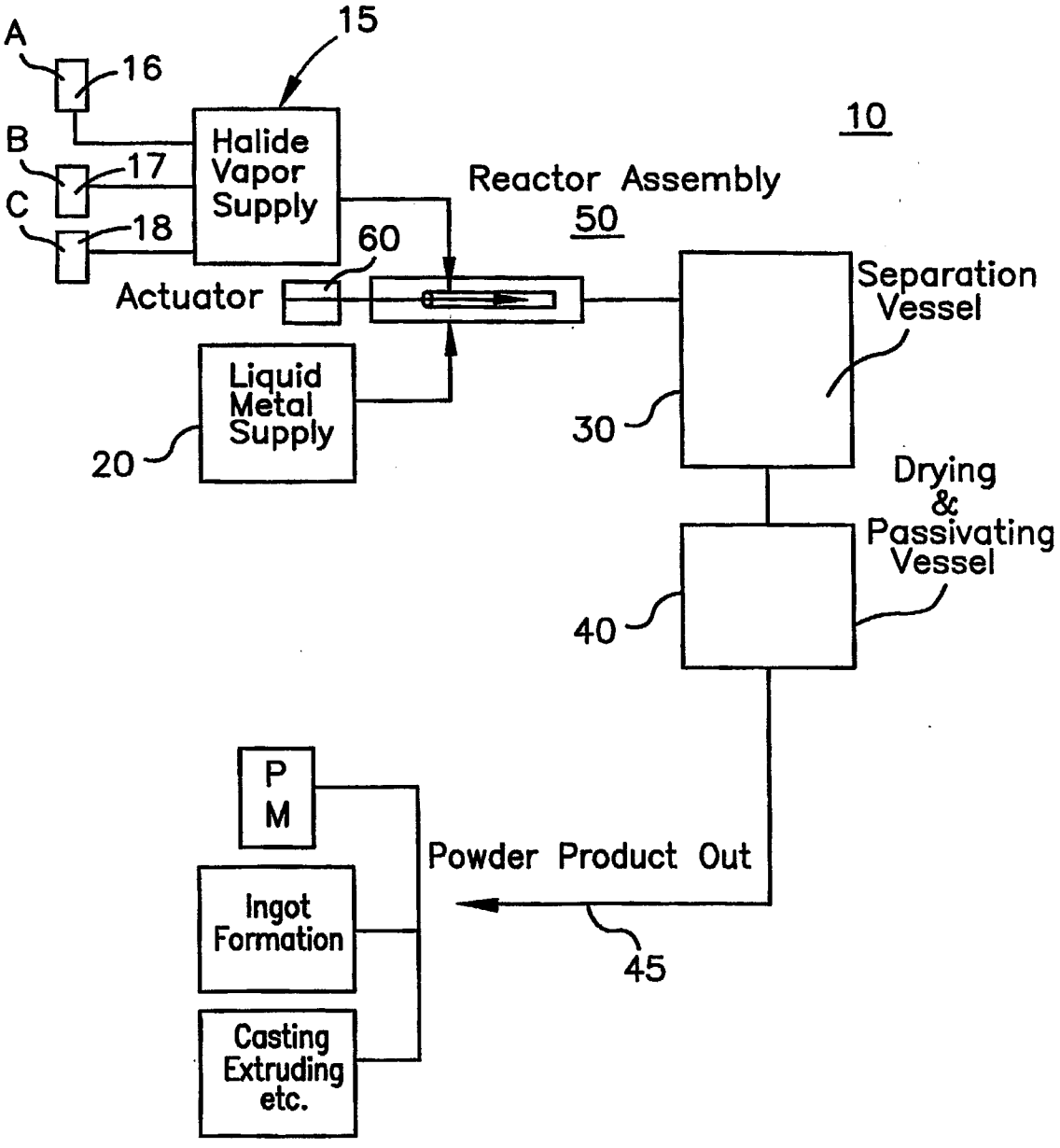
A
16
15
B
17
C
18
Halide Vapor Supply
10
Reactor Assembly
50
60
Actuator
Separation Vessel
Liquid Metal Supply
20
30
Drying & Passivating Vessel
40
P M
Powder Product Out
Ingot Formation
45
Casting Extruding etc.

FIG. 1

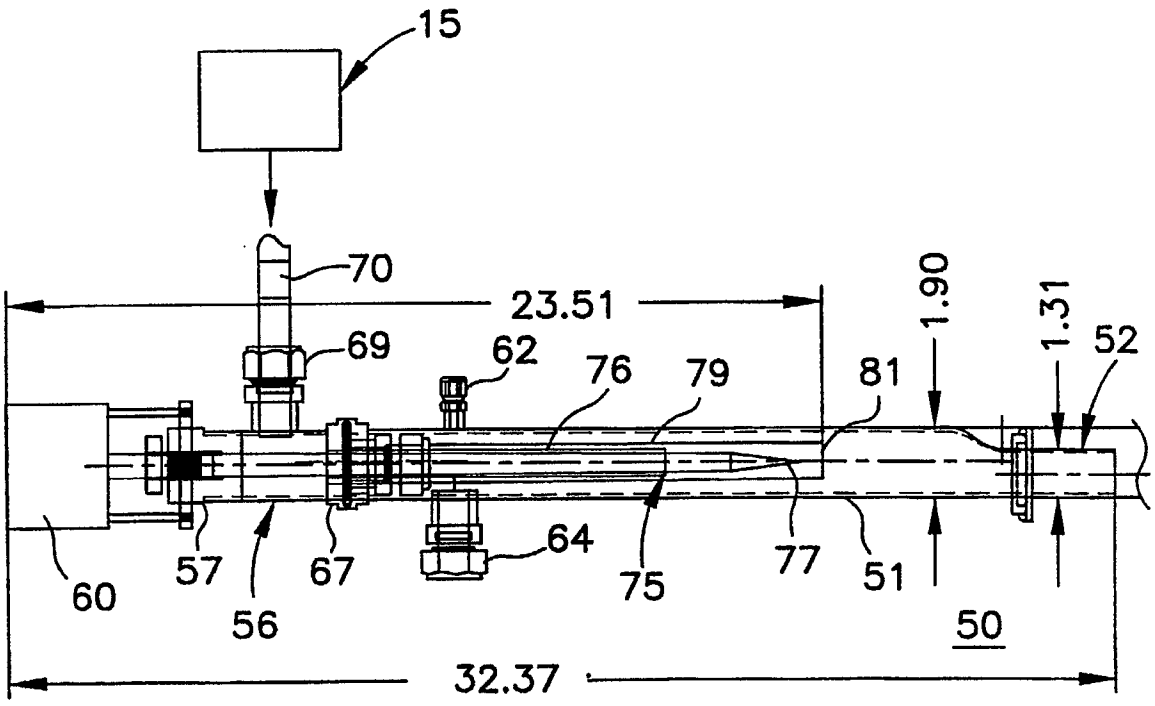

FIG. 2

INTERNATIONAL SEARCH REPORT

Internati pplication No
PCT/US 03/27650

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C22B34/12 B22F9/28

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C22B B22F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 958 106 A (ANDERSON RICHARD PAUL ET AL) 28 September 1999 (1999-09-28) cited in the application	1
X	abstract	13,14

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
15 December 2003	15/01/2004

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31–70) 340–2040, Tx. 31 651 epo nl, Fax: (+31–70) 340–3016	Alvazzi Delfrate, M

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Information on patent family members

Internati[illegible]pplication No
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